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# Effect of Sonication Parameters on Mechanical Properties of *In-situ* Amine Functionalized Multiple Layer Graphene/Epoxy Nanocomposites

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In this investigation, combined effects of various sonication time and power on dispersion of 0.75 wt.% In-situ amine functionalized multi-layer Graphene (AF-MGL) in polymer matrix were studied. To ensure proper dispersion of AF-MGL in the epoxy matrix, sonication times of 10, 40, 70 min and powers of 20, 40, 60 W were used. The tensile test results indicate an initial increase in tensile modulus at smaller sonication time and then decrease for more sonication times. The highest tensile modulus and tensile strength were obtained at 40 W, 40 min and at 20 W, 40 min respectively. In order to classify the AF-MGL dispersion status, a scanning electron microscope (SEM) was used. With increased sonicity time and strength, dispersion has been noticed.

**Keywords:** Composites, Epoxy, Functionalization, Graphene, Microscopy

## Introduction

Graphene as an allotrope of carbon, two-dimensional (2D) nature and its unprecedented properties has become the most studied nanomaterial in the area of polymer nanocomposites since their discovery in 2004.[1,2] Because of excellent mechanical properties and abundant explicit surface area, graphene based polymer nanocomposites can be regarded with significantly enhanced mechanical properties compared to neat epoxy matrix material. This includes a good execution of the critical dispersion problem. To develop better quality nanocomposites, breaking-up of the agglomerates and uniform dispersion of the exfoliated graphene into polymeric matrix are needed.[3–6]

Many researchers have made several efforts for improving the dispersibility of graphene into polymer matrix with the help of three methods namely physical dispersion approaches viz. water bath or probe sonicator based ultrasonication[7–9], shear mixing into solvent[10], covalent bonding approaches normally obtained by solution of graphene oxide and organic small molecules, polymers, or other materials with outstanding emulsifiable by some chemical reactions[11,12]and non-covalent bonding approaches viz. Chemical functionalization, to create surface functionalities to make graphene more chemically compatible with matrix which is attributed to improved dispersion.[13–15]

The main challenge of graphene dispersion into polymer matrix is owing, its small size, insolubility and vander Waals forces due to which graphene is prone to agglomeration. Sonication is popular approach to address the mixing and dispersion of nanomaterials with the help of highly intensive acoustic energy. Sureties *et al.* observed that as the sonication time increases, the tensile strength of graphene enhance becomes more important, for sonication time 60 min taking its maximum value.[16–18]

The goal of research was to analyze influence of dispersion quality on ultimate mechanical properties of *in-situ* amine multi-layer functionalized graphene (AF-MGL)/epoxy nanocomposites that were inspired by sound parameters. In order to achieve this objective, multiple sonic times and power output were selected to generate multiple AF-MGL dispersion conditions. SEM has been investigated for the consistency of dispersion of AF-MGL particles in the matrix.

## Materials and Methods

### Materials

The materials obtained in the Sakshi Dyes & Chemicals, New Delhi study included low viscosity of the bisphenol epoxy di glycidyl ether LY-556,

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thermoelectric polymer, with high adhesion properties, thermal stability , high modulus and high intensity of corrosion as well as high interconnection properties, coated with Triethylene-tetra-mines (TETAs), HY-951 with 100/10 mixing ratios. In-situ amine worked on several layers of Graphene from Platonic Nanotech Pvt, with lengths between 5–25 µm and diameters between 15 and 25 nm. Ltd, it's been used by Jharkhand.

#### Preparation of nanocomposites

Initial hand mixing of around 25 g epoxy suspension and 0.75 wt. percent AF-MGL started the manufacturing process. Then effects of these parameters on manufactured nanocomposites were investigated by sonication at different output power (20, 40 and 60 W), and durations (10, 40 and 70 min). The hardener was added and mixed by casting in steel moulds after the dispersion of AF-MGL through sonification. The samples were cured at room temperature followed by post cure at 90°C for 2 hours and at 100°C for 1 hour.

#### Mechanical testing and characterization

A room temperature D638-10 with the MTS Universal Testing Machine (MTS-610 with Static tension Cell of Capacity 10 KN) was used for tensile testing. The tensile power, elongation and modulus value were recorded by an extensometer (MTS Advantage AHX850 dynamic extendometer with a 12.5 mm gauge length). At crosshead speed of 1.5 mm/min samples were loaded to fail. This very low charge rate was chosen because nanocomposites were fragile in nature.

By using SEM, the fracture surface analysis was performed (JEOL JSM-6010LA).

## Results and Discussion

#### Tensile and Fracture Behavior

Measured tensile properties (strength and elastic moduli) of AF-MGL have effect of sonic parameters which is shown in Fig. 1, 2 and Table 1. In general, the tensile strength shows an upward trend at output power of 20 W and 60 W, hitting peak, and then decreasing, by increasing the sonication time. The tensile strength trend for 40 W, on other hand, is growing.

Comparing tensile strength of the specimens manufactured at different strength (20, 40 and 60 W) for a constant period of 10 minutes, the effective dispersion of AF-MGL is only achieved at less power (20 W) and low time (10 minutes), which is a key to enhance in mechanical properties. Soning for longer times increases dispersion, resulting in largest amount of tensile strength. Higher energy suspension is equal to more efficient and longer a time that leads to better dispersion, though the AF-MGL aspect ratio must eventually be reduced. The longer the sonication duration influences the dispersion positively. At all different sonic forces and periods, the traction intensity of processed Nanocomposites is stronger than that of tidy Epoxy. As evident from Table 1 biggest gains in tensile strength is 40 minutes sonication at 20 W, up to 14.3%. The findings also show that the tensile strength is lower at about 1.64% when 10 minutes at 20 and 40 W are sonically enhanced.

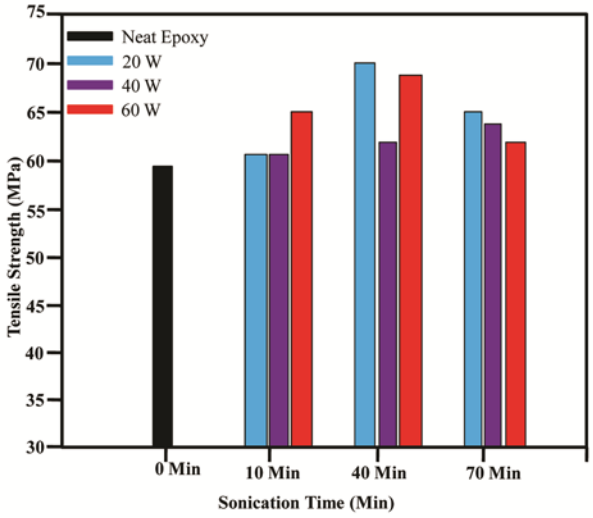


Fig. 1— Tensile strength vs. sonication time and power

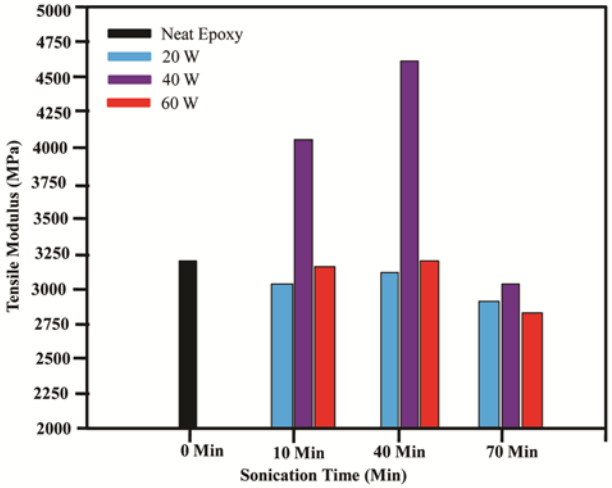


Fig. 2 — Bar graph tensile modulus vs. sonication time and power

Table 1 — Tensile strength and tensile modulus of AF-MGL / epoxy nanocomposites fabricated at different sonication powers and durations

| Power (W) | | 20 | | | 40 | | | 60 | | |
|---|---|---|---|---|---|---|---|---|---|---|
| Tensile Properties | Neat Epoxy | Sonication | Time | (Min) | Sonication | Time | (Min) | Sonication | Time | (Min) |
| | | 10 | 40 | 70 | 10 | 40 | 70 | 10 | 40 | 70 |
| Strength (MPa) | 60 ± 1 | 61±1 | 70±1 | 65±2 | 61±1 | 62±1 | 64±1 | 65±2 | 69±2 | 62±1 |
| Strength Increase (%) | — | 1.64 | 14.3 | 7.69 | 1.64 | 3.23 | 6.25 | 7.69 | 13.04 | 3.23 |
| Tensile Modulus (MPa) | 3250 ± 100 | 3019±160 | 3132±135 | 2790±160 | 4019±120 | 4729±135 | 3060±110 | 3142±110 | 3250±150 | 2979±90 |
| Modulus Increase (%) | — | −7.65 | −3.77 | −16.49 | 19.13 | 31.28 | −6.2 | −3.44 | 0 | −9.1 |

At different periods of sonification and production, demonstrates the Young's module of nanocomposites. The tensile modulus is reduced by 20 W and 60 W relative to the smooth epoxy. Sonicity for 40 min at 40 W contributes to a 31.28 percent higher tensile module than the clean epoxy. The tensile module shows an upward trend at all sonic power levels, with an increase of the suction time, to a limit, and then drops. Sonication for 70 minutes tends not to be a good time to generate high modulus nanocomposites. The Fig 1 and Table 1 Both in depth display the results. In addition to a long sonic duration, very high and low sonic powers are not appropriate for producing high modulus nanocomposites.

The agglomerations function as tension thresholds and hence the initiation of a crack. SEM micrograms of tensile specimens' broken surface indicate that the cracks are usually the source of agglomeration. Higher dispersion contributes to lower agglomeration and smaller average size of less stressful agglomerates. Best dispersion is found at 20 and 60 W after 40 min, according to results provided in Table 1. Increasing the sonic time causes decrease of the interposition of AF-MGL in the epoxy matrix and thus more dispersion.

SEM was used in the control and examination of nanocomposites sample fracture surfaces. Surface fracture were tested with tidy epoxy and its nanocomposites, at constant power produced with 40 W but sonication over different times (Fig. 3a–c). As seen, the increase in sonic time contributes to better AF-MGL dispersion, (Fig. 3 b) means that AF-MGL is totally improved.

The effect of the sonic power of dispersion performed over a short duration can be differentiated using Fig. 4 (a) and (b). Comparing the sonication results with a potential of 40 and 60 W for 10 minutes supports the hypothesis that certain agglomerates are present inside the specimens and that the modulus increases. The Young Nano-composite Module (10 min, 40 W) increased 19.13 percent compared to

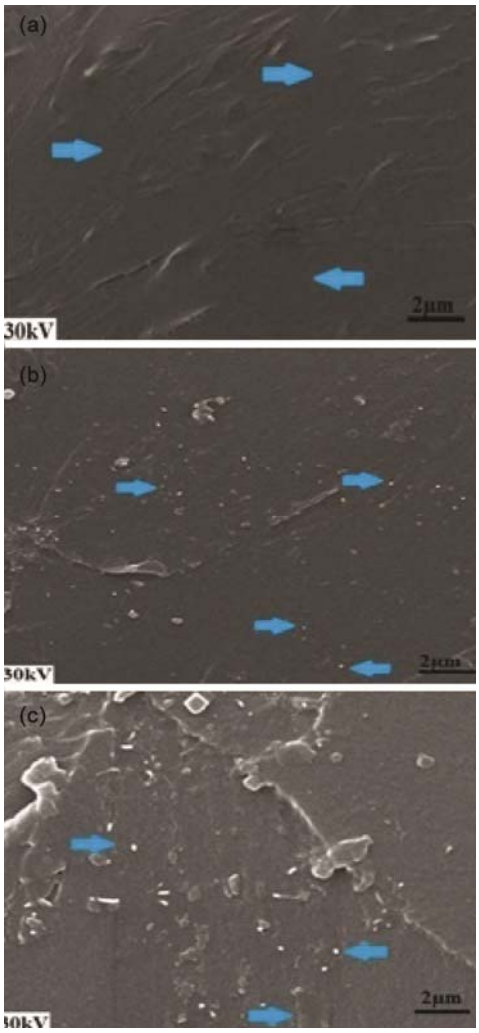


Fig. 3 — SEM micrographs of fracture surface of neat epoxy (a) dispersion of AF-MGL in epoxy; 40 min (b) and 60 min (c) at power sonication of 40 W

the Epoxy module according to the Table 1. In fact, Young's modulli bases not on AF-MGL aspect ratio but on the dimension and distribution of agglomerates. No agglomerate is visible at 60 W (Fig. 4b), while agglomerates are seen in the Fig. 4(a). The Young modulus has been decreased by 3.44% relative to epoxy.

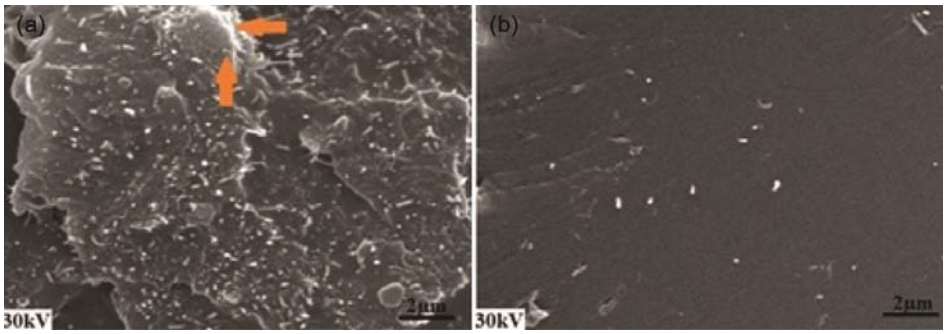


Fig. 4 — SEM micrographs of the dispersion of AF-MGL in epoxy; 10 min at 40W (a) and 10 min at 60 W (b)

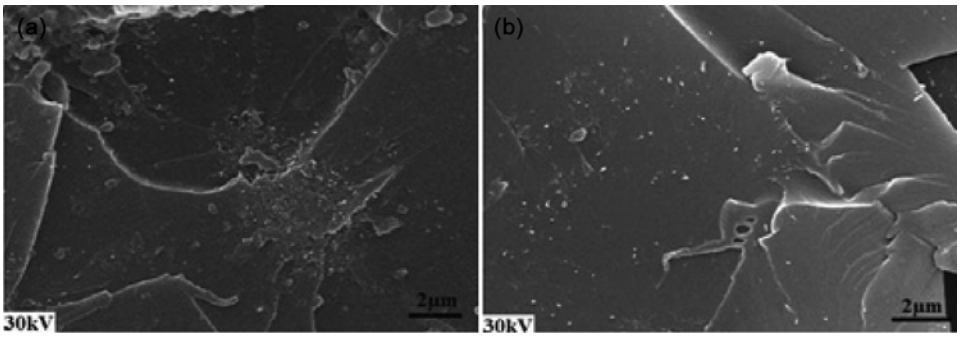


Fig. 5 — SEM micrographs of dispersion of AF-MGL in epoxy; 40 min at 20W (a) and 40 min at 60 W (b)

The sonic power effect can be used to equate two separate higher and lower power and more duration outputs with higher duration as shown in Fig. 4 (a) and (b). At power of 60 W and 40 min, better emulsifiable is found, as shown in Fig 5. For more emulsifiable reasons nanocomposites tensile strength to grow to 61 MPa in 20 W and 65 MPa in 60 W.

## Conclusions

Ultrasonication was used as conventional dispersing technique in this study and on tensile properties of AF-MGL / epoxy nanocomposites were investigated sonication output powers and time duration. In order to research how they can affect dispersion state of AF-MGL in matrix, different output powers and times have been applied. The emulsifiable of AF-MGL in matrix was analyzed using SEM micrographs. The results of tensile test showed that tensile strength was first enhanced by increasing sonicity time by 20 and 60 W and then decreasing to around 14.3% higher than that of clean epoxy. For the tensile module, a trend was observed in every power. For intermediate length and power levels, the best tensile strength results were achieved. Results from the SEM images show that changes in AF-MGL scatter status have caused tensile properties to be changed. The improved sounding power and time have resulted in better dispersion, although the aspect ratio of AF-MGL has been reduced.

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